

# Thermal Diffusivity Measurement of Glass at a High Temperature by Use of the Flash Method

Thomas KABAYABAYA, Xinxin ZHANG and Fan YU  
Department of Energy Engineering, School of Mechanical Engineering  
University of Science and Technology Beijing, 100083 Beijing, China  
xxzhang@me.ustb.edu.cn

**Abstract** A measurement of the Thermal Diffusivity of a solid semi-transparent material from the range of ambient temperature to a high by the use of the Flash Method is investigated in the present paper. A semi-transparent layer, which emits, absorbs but does not scatter, is considered. The problem is supposed to be linear. A one-dimensional transient energy equation transfer by conduction and radiation for a finite medium is solved. The analytical solution is obtained by considering some mathematical approximations, using the Laplace Transform, and the kernel substitution technique. The experimental design presented in this work is an original technical concept, which enables a significant reduction of contact heat loss between the sample and its sample holder. It also eliminates heat loss between the sample and detector, and increases the speed and precision of experimental measurements. The experiments are done on different kinds of glass with different boundary conditions and different thicknesses. Heat losses on both the front face and rear face of the sample are taken into account. A very simple model based on the quadrupole method is used to theoretically determine the thermal diffusivity of the semi-transparent material by taking into account both conduction and radiation. This model allows the use of very short resulting computation times, and clarifies the consideration of heat losses on the two faces of the sample. The theoretical results are found to be in agreement with the experimental results.

## 1. Introduction

For many decades, the needs in science and technology have pushed many authors to develop new methods to determine thermal properties of materials with more accuracy. The most preferred of them is the flash method conceived for the first time by Parker <sup>[1]</sup>. The flash method is an impulse method for which, the front face of the sample is subjected with a short thermal impulse, and then, an analysis of the evolution of the temperature versus time (thermogram) on the rear face of the sample is done. It was developed in order to eliminate the problems of thermal contact resistance between the specimen and its associated heat source, and to minimize the heat losses by making the measurements in a time short enough so that very little cooling could take place. Many experiments to improve the flash method have been developed <sup>[2-8]</sup>. Some of them are to determine thermal diffusivity by considering several points of the thermogram or its all significant part, and by taking into account heat loss on the two or three faces of the sample <sup>[9-12]</sup>. A technique of partial temporal moment of the order 0 and  $-1$  issued from the experimental thermogram

and the model, which regroups the advantages of the above mentioned two techniques was developed<sup>[13]</sup>.

The flash method has been extended to study thermal properties of semitransparent materials for various boundary conditions<sup>[14-15]</sup>. Several authors solved the problem of one-dimensional transient heat transfer by coupled conduction-radiation for a finite medium<sup>[16-21]</sup>. An experimental device, suitable for the transient measurement of the phonic diffusivity of semitransparent material in the range from 300 to 800 K, was produced<sup>[18]</sup>. The phonic diffusivity was directly extracted from the measured parameter in an identification process for opaque materials<sup>[22, 23,30]</sup>. A theoretical model of one-dimensional transient combined heat transfer for different thermal responses with different experimental conditions was developed<sup>[19]</sup>. Simulations have shown that for conditions of small equivalent optical thickness, and in the case of reflecting walls, the flash method provides a direct measurement of the phonic diffusivity of glass in the same way as it does for opaque materials. An exact analytical solution was found with a linear transfer assumption, using a kernel substitution technique<sup>[24]</sup>. This technique demonstrates that the Rosseland gray coefficient<sup>[25-27]</sup> has to be used in the gray absorbing model in order for non-gray behavior to be described correctly. The quadrupole representation of the problem<sup>[28, 12 - 13]</sup> was used in order to facilitate the resolution.

The present work is concerned with the thermal diffusivity measurement of glass at high temperature by means of the flash method. Physical and theoretical models are first defined, and an original technical concept, which enables to minimize heat loss during the experiments, is presented. This new experimental device, not only eliminates the problems of thermal contact resistance between the specimen and its associated heat source, but also eliminates the thermal contact resistance between the specimen and the detector. The thermal contact between the sample and sample holder is reduced to a minimum. Theoretical results show the effect of the absorption coefficient and the thickness of the sample on heat transfer in the semi-transparent medium. Experimental results are presented.

## **2. Physical and Theoretical Models**

### **2.1 Physical model**

The problem under consideration refers to the hypotheses<sup>[16, 29, 31-33]</sup>: (1) finite medium; (2) emitting, absorbing but non-scattering medium; (3) gray medium (both the absorption coefficient and the refractive index are independent of wavelength); (4) opaque boundaries with diffuse gray emissivities and reflectivities; (5) the medium is initially at uniform temperature  $T_0$  and receives a quantity of heat  $Q$  at time  $t>0$ ; (6) temperature from the room temperature to the sample's melting temperature; (7) heat losses on the sample surfaces are considered.

### **2.2 Theoretical Model**

A theoretical model of one-dimensional transient combined conduction-radiation heat transfer is considered. The transient behavior of temperature within a homogenous, isotropic slab can be found by solving the following energy equation<sup>[16-17]</sup>:

$$\rho C_p \frac{\partial T}{\partial t} = -\frac{\partial q_c}{\partial z} - \frac{\partial q_r}{\partial z} \quad (0 < z < e, \quad t > 0) \quad (1)$$

where,  $q_c = -k_{ph} \partial T(z,t)/\partial z$  is the purely conductive heat flux, and  $q_r$  is the purely radiative heat flux.

The following dimensionless variables are introduced for the best calculation:

- Dimensionless temperature:

$$\theta = T^* - T_0^* = (T - T_0) / (Q / \rho C_p e)$$

where  $(Q / \rho C_p e) = T_{ad}$  is the adiabatic temperature,  $Q$  is the energy density received by the layer,  $\rho$  is the density,  $C_p$  is the specific heat,  $e$  is the slab thickness, and  $T_0$  is the reference temperature.

- Dimensionless time variable:

$$t^* = at / e^2$$

It is also called the Fourier number based on the phonic diffusivity  $a$  of the semi-transparent layer.

- Dimensionless space variable:

$$z^* = z / e.$$

- Optical thickness:

$$\tau_0 = \chi e$$

where  $\chi$  is the absorption coefficient

- Conduction-to-radiation parameter:

$$N_{pl} = k_{ph} \chi / 4n^2 \bar{\sigma} T_0^3.$$

It is also called Planck number or Stark number.

- Dimensionless net radiative heat flux:

$$q_r^* = q_r / 4n^2 \bar{\sigma} T_0^4.$$

- Dimensionless intensity (or incident radiation):

$$I^* = \pi I / 4n^2 \bar{\sigma} T_0^4.$$

Applying the above dimensionless variables, Eq. (1) becomes:

$$\frac{\partial^2 \theta}{\partial z^{*2}} - \frac{\tau_0 T_0}{N_{pl}} \frac{\partial q_r}{\partial z^*} = \frac{\partial \theta}{\partial t^*} \quad (0 < z^* < 1, \quad t^* > 0) \quad (2)$$

Assuming no spectral properties, the dimensionless radiative heat flux can be expressed as [18-21].

$$q_r(z^*) = 2 \left[ I^+(0) E_3(\tau_0 z^*) + \frac{\tau_0}{4} \int_0^{z^*} \left( 1 + \frac{\theta(z')}{T_0} \right)^4 E_2(\tau_0(z^* - z')) dz' \right] \\ - 2 \left[ I^-(1) E_3(\tau_0(1 - z^*)) + \frac{\tau_0}{4} \int_{z^*}^1 \left( 1 + \frac{\theta(z')}{T_0} \right)^4 E_2(\tau_0(z' - z^*)) dz' \right] \quad (3)$$

In Eq. (3), the dimensionless intensities given by the radiative limits under consideration  $I^+(0)$  and  $I^-(1)$  are defined as follows:

$$I^+(0) = \frac{a_1 + b_1 a_2}{1 - b_1 b_2} \quad \text{and} \quad I^-(1) = \frac{a_2 + b_2 a_1}{1 - b_1 b_2} \quad (4)$$

with

$$a_1 = \frac{\varepsilon_1}{4} \left( 1 + \frac{\theta(0)}{T_0} \right)^4 + 2\rho_1 \int_0^1 \frac{\tau_0}{4} \left( 1 + \frac{\theta(z')}{T_0} \right)^4 E_2(\tau_0 z') dz' \\ a_2 = \frac{\varepsilon_2}{4} \left( 1 + \frac{\theta(1)}{T_0} \right)^4 + 2\rho_2 \int_0^1 \frac{\tau_0}{4} \left( 1 + \frac{\theta(z')}{T_0} \right)^4 E_2(\tau_0(1 - z')) dz' \\ b_i = 2\rho_i E_3(\tau_0) \quad (\text{for } i=1,2)$$

where  $\varepsilon_i$  and  $\rho_i$  represent gray diffuse emissivity and gray diffuse reflectivity respectively. In the case of opaque boundaries, we have  $\varepsilon_i + \rho_i = 1$ .

The semi-transparent material is always considered in a perturbed state where the deviations from a reference state of radiative equilibrium are small. Thus, temperature  $T$  at any location in the semi-transparent material sample will be equal to  $T_0 + \theta$  where  $T_0$  is the reference temperature and  $\theta$  a small perturbation. According to the dimensionless variables introduced above, the assumption

$$\left( 1 + \frac{\theta}{T_0} \right)^4 \approx 1 + 4 \frac{\theta}{T_0}$$

is valid. The kernel substitution technique <sup>[5]</sup> shows that, in Eq. (3),  $E_2(z) \approx m \exp(-bz)$ ,

and  $E_3(z) \approx (m/b) \exp(-bz)$ , with  $m = 3/4$  and  $b = 3/2$  respectively.

Applying this kernel substitution and the assumption given above, the radiative heat flux Eq. (3) is expressed as:

$$q_r(z) = I^+(0) e^{-\tau z^*} - I^-(1) e^{-\tau(1-z^*)} - \frac{1}{4} (e^{-\tau z^*} - e^{\tau(1-z^*)})$$

$$+ \frac{\tau}{T_0} \int_0^{z^*} \theta(z') \exp(-\tau(z^* - z')) dz' - \frac{\tau}{T_0} \int_z^1 \theta(z') \exp(-\tau(z' - z^*)) dz' \quad (5)$$

Here the variable  $\tau = 3\tau_0/2$  results from the kernel substitution and is introduced for convenience. For the same reason,  $N_{pl}$  becomes  $N = 3N_{pl}/2$ . When differentiating the Eq. (2) twice, one has:

$$\frac{\partial^4 \theta}{\partial z^{*4}} - \frac{\partial^3 \theta}{\partial z^{*2} \partial t} = \frac{\tau T_0}{N} \frac{\partial}{\partial z^*} \left( \frac{\partial^2 q_r}{\partial z^{*2}} \right) \quad (6)$$

The substitution of the second order derivative of the radiative heat flux Eq. (5) in Eq. (6) yields:

$$\frac{\partial^4 \theta}{\partial z^{*4}} - \frac{\partial^3 \theta}{\partial z^{*2} \partial t} = \frac{\tau^3 T_0}{N} \frac{\partial q_r}{\partial z^*} + 2 \frac{\tau^2}{N} \frac{\partial^2 \theta}{\partial z^{*2}} \quad (7)$$

At this point, the expression  $\partial q_r / \partial z^*$  appears along with partial derivatives of  $\theta$ , as in equation (2). Thus introducing Eq. (2) in Eq. (7) and applying Laplace transform in time.

$$\bar{\theta}(p) = \int_0^\infty \theta(t) \exp(-pt) dt \quad (8)$$

We have the following ordinary differential equation in the Laplace domain:

$$\frac{d^4 \bar{\theta}}{dz^{*4}} - (p + 2 \frac{\tau^2}{N} + \tau^2) \frac{d^2 \bar{\theta}}{dz^{*2}} + p \tau^2 \bar{\theta} = 0 \quad (9)$$

Eq. (9) is solved by the use of the quadrupole method <sup>[21]</sup>. A linear relationship between Laplace transformations of temperature-heat flux,  $\bar{\theta}(0)$  and  $\bar{\phi}(0)$  at the front side of the sample, and  $\bar{\theta}(1)$  and  $\bar{\phi}(1)$  at the rear face is provided by a transfer matrix of the quadrupole. Considering equal heat transfer coefficients,  $h$  on both sides of the sample, the problem is expressed in the following matrix:

$$\begin{bmatrix} \bar{\theta}(0) \\ \bar{\phi}(0) \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ H & 1 \end{bmatrix} \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} 1 & 0 \\ H & 1 \end{bmatrix} \begin{bmatrix} \bar{\theta}(1) \\ \bar{\phi}(1) \end{bmatrix} \quad (10)$$

where  $H = h e / k_{ph}$  is the Biot number. The Laplace transformation of the temperature at the rear face of the sample is:

$$\bar{\theta}(1) = \bar{\phi}(0) / [H(A + D + BH) + C] \quad (11)$$

### 3. Simulation results and discussion

The rear-face thermal behavior on three different kinds of sample glass, black boundaries and reflecting boundaries, are simulated at a high temperature (900°C). The three considered kinds of glass have Rosseland absorption coefficients  $\chi_{R1}=49.5 \text{ m}^{-1}$ ,  $\chi_{R2}=250 \text{ m}^{-1}$ , and  $\chi_{R3}=2500 \text{ m}^{-1}$  respectively. The theoretical results of coupled heat transfer for three different kinds of sample glasses with black boundary conditions (Figs.1 and 2), and reflecting boundary conditions (Figs.3 and 4) are illustrated.

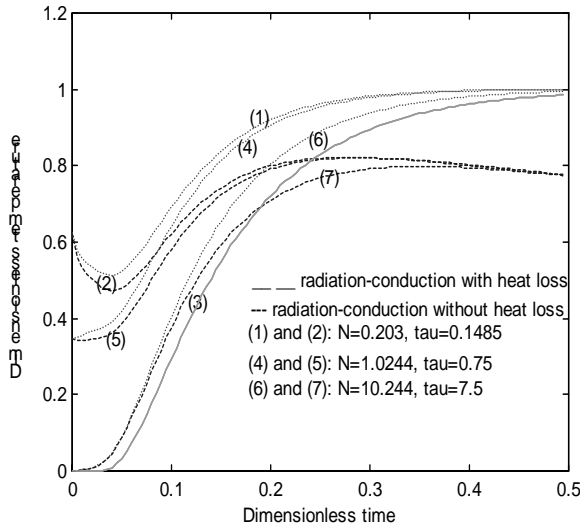


Fig.1. Rear face thermal responses of three different samples with black boundaries ( $e = 0.002\text{m}$ )

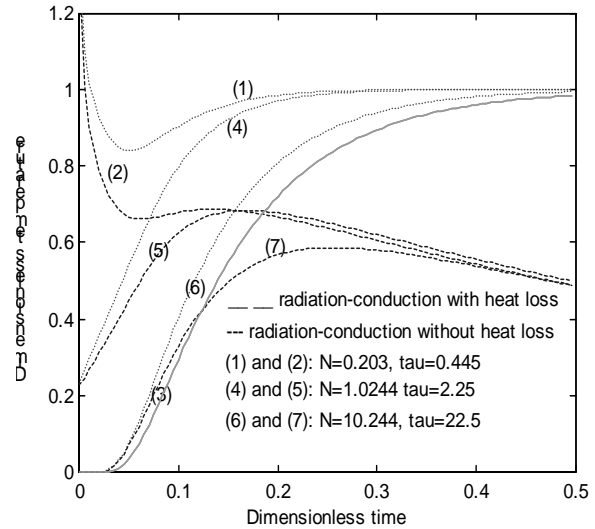


Fig.2. Rear face thermal responses of three different samples with black boundaries ( $e = 0.006\text{m}$ )

For all figures (Figs.1 – 4), the curves 1 and 2 are the thermal response on the rear face of the first kind of sample glass ( $\chi_{R1}=49.5 \text{ m}^{-1}$ ); the curves 4 and 5 are the thermal response on the rear face of the second kind of glass ( $\chi_{R2}=250 \text{ m}^{-1}$ ); the curves 6 and 7 are the thermal responses on the rear face of the third kind of glass ( $\chi_{R3}=2500 \text{ m}^{-1}$ ), and curve 3 represents the rear face thermogram for the case of pure conduction.

The comparison made for Figs. 1 and 2 shows that for the case of black boundaries, the thermal response differs largely from the classical thermogram (case of pure conduction). As it has been reported in <sup>[19]</sup>, the initial peak, which appears at a very short time is caused by a direct energy exchange between the two black boundaries through the medium of small radiative resistance. The rear face behaves as if it is the perturbed surface. The temperature decreases, passes through a minimum and increases under the progressive influence of a heat flux connected to the coupled mechanism of both conductive and radiative transfer. On both Fig.1 and 2, the radiative effects are important for the sample

glass with a small absorption coefficient (curves 1 and 2) than for the sample with high absorption coefficient (curves 4 and 5). Consider now Fig.1 (curves 1, 2, curves 4, 5 and curve 6,7) and Fig.2 (curves 1, 2, curves 4, 5 and curves 6, 7). It is clear that, for three sample glasses of the same kind, but with different thicknesses, the radiative effects and heat losses are more significant for the sample with the greater thickness.

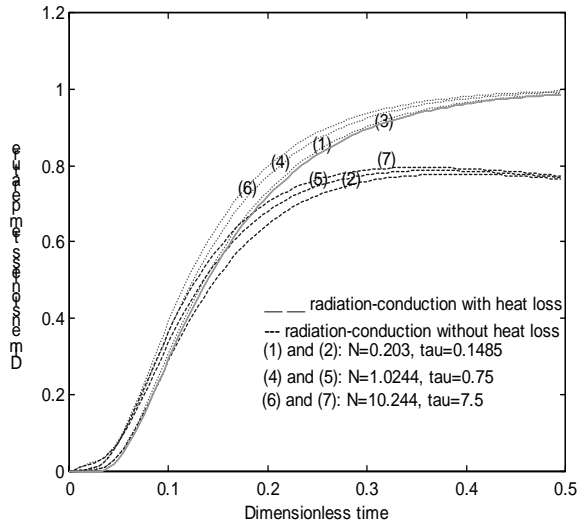


Fig. 3. Rear face thermal responses of the three different kinds of samples with reflecting boundaries ( $e=0.002\text{m}$ )

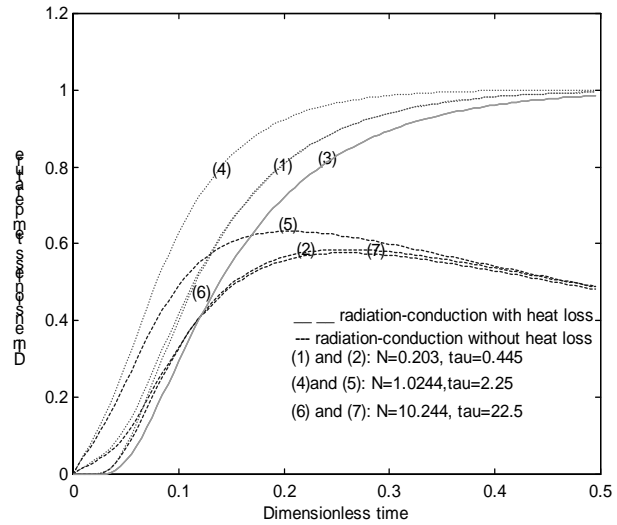


Fig.4. Rear face thermal responses of the three different kinds of samples with reflecting boundaries ( $e=0.006\text{m}$ )

In Figs.3 and 4, the case of reflecting boundary conditions for two above kinds of glass is studied. The thermograms obtained here behave as a classical thermogram. Consider the same comparisons of different curves with curve 3 (case of pure conduction) as we did for the case of black boundaries in order to identify the radiative effects on each measurement for every considered sample. One can conclude that, for the case of reflecting boundaries an increase of the sample thickness increases the radiative effects and heat loss as it is for the case of black walls. But here we see that for glasses with great thickness the radiative effects are not significant for sample glasses which have very high or very small absorption coefficients (curves 1, 2, 4, 5, 6 and 7 of Fig.4). We can also say that, if the absorption coefficient is very small (curve 1 of Fig.3), the radiation can be neglected.

#### 4. Experimental measurement

The experimental setup is illustrated in Fig.5. The sample is placed into the furnace in a determined position so that the heat flux from laser source may be pointed perpendicularly to its front face. The duration of the heat impulse is around 1ms. With a

pump, a vacuum is created in the furnace in order to avoid heat losses by convection at high temperatures and to allow the setting of the sample at a stable temperature. The data of temperature rise at the rear face of the sample, and the temperature in the furnace are collected by means of the thermal couples Chrome/Aluminum thermal couple and led to the monitor for their analysis. One has to mention that, during the experiment, the temperature of the laser source is permanently moderated by cool water.

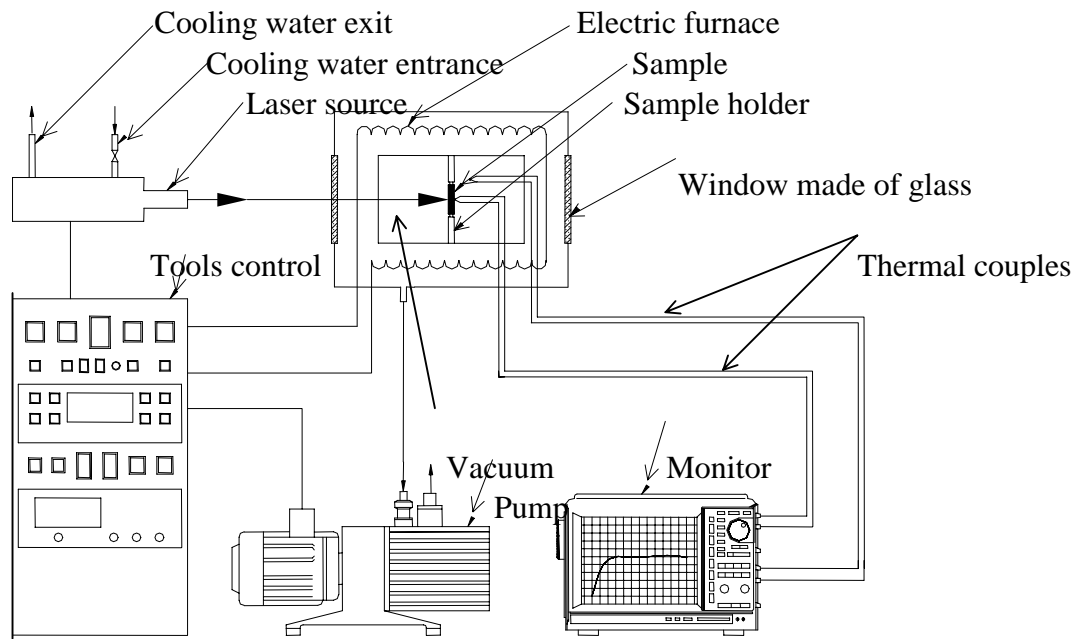


Fig. 5 Experimental setup

The simple method used to determine the thermal diffusivity of solid materials is Parker's method. But this method is efficient only for the determination of thermal diffusivity of opaque material, and in the case where the heat losses are not considered.

For the present work, where the study is led on semi-transparent material at high temperature, one has to take into account both conduction and radiation. Heat losses are also considered. The measurements are done on the silicate glass with reflecting boundaries. The measurement results are shown on the Figs.6 and 7.

## 5. Conclusion

A new experimental technique design for the measurement of the thermal properties of semi-transparent materials by the Flash Method at high temperature was conceived. The advantages of this technique over the others already used by many authors is that it can reduce the heat loss to a minimum temperature. A theoretical model of a 1D transient heat transfer has been developed.

The influence of the thickness and boundary conditions for different kind of glasses were discussed. It was shown that, for the case of a black wall, the radiative effects are important for the glass with a small absorption coefficient, and they increase with the thickness of the sample. For the case of reflecting boundaries, the radiative effects are more important for the sample with a high absorption coefficient and they increase with thickness.

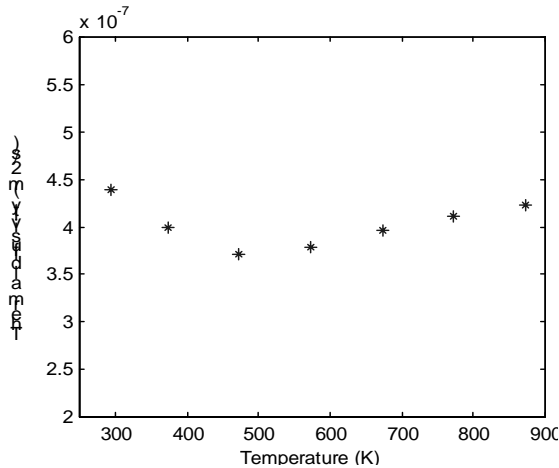


Fig.7: Thermal diffusivity of silicate glass as function of temperature (e = 2mm)

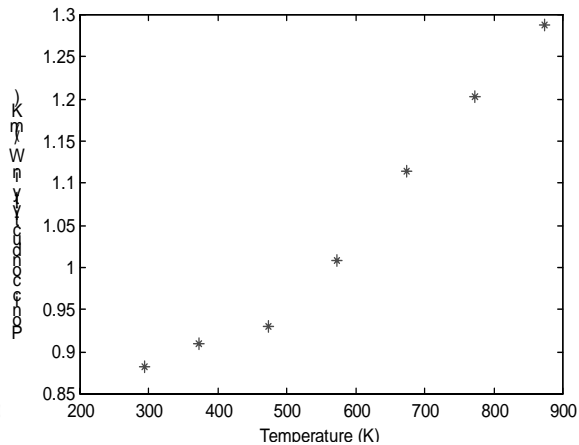


Fig.8. Phonon Diffusivity of silicate glass as function of temperature (e=2mm)

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